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Fiber Glass Pulling

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INTRODUCTION

Materials processing in space has offered to many researchers the opportunity to perform many types of reactions and processes in either low gravity or in a containerless environment. Of the many research activities carried out in the various NASA facilities such as Spacelab , the KC-135 aircraft, and at the Drop Facilities at Marshall Space Flight Center; very little research has really been done on glass fiber processing. In spite of the infrequent experimentation performed, some interest has been shown by a number of scientists. Most notably Dr. Ed Etheridge at MSFC who sponsored this work.

A commercial driver for activity in the area of fiber glass is the use of low attentuation fiber optics for communications and other purposes. In some respects the containerless processing environment has been considered ideal for the manufacturing of glass fibers. However, depending upon the source of the conjectures, some predict either less crystalization may occur in low gravity and others predict lesser amounts of contaminants will occur if processed containerlessly. Subsequently some effort has been underway over the last few years to demonstrate the feasibility of pulling fibers in space or to determine through intermediate steps that such space experiments are worthwhile. A

number of technological problems do exist in such experiments. This report concludes one such study as an intermediate step and will try to identify the problem areas which were experienced in this effort and will need to be overcome for space based experiments to be successful.

The experimental aspects of this work concentrated on acoustic levitation as the primary technique to accomplish containerless processing on nonconducting materials in space. Consequently previous research at the Jet Propulsion Laboratory and Marshall Space Flight Center indicated that acoustic levitation furnaces would certainly be viable technique for fiber-pulling in space. In terms of the materials most appropriate for fiber pulling experiments, the heavy metal fluorides seemed to have the most promise in terms of product newness and potential need in the communications industry s the lowest attenuation fibers currently known.

Heavy Metal Fluorides

Several compendia have been published in the last few years on the research interests of metallic fluorides for glass fibers and their potential as a medium for long-distance communications. The chapter by Drexhage(1) is excellent in reviewing the entire

field. Calculations from Miyashita and Manabe (2) have indicated that the heavy metal fluorides may provide the least attenuation factor of any of the materials currently being considered for fiberoptic communications. Unfortunately the optical transmission window for these glasses occur at longer wavelengths (1 - 5 microns) than the corresponding silicate fibers. As a result the marketplace has lesser availibility of transmitters and receivers for these fibers. The increased need for such electronic systems has not matured in the same rate as it has for silica fibers. Commercially available fluoride fibers are now entering the marketplace and will certainly provide incentives to develop the accompanying telecommunication devices.

Figure 1 shows the theoretical model for transmissive behavior of fluoride glasses (2). The infrared transmission is limited on the long wave-length side by multi-phonon scattering and on the short wave-length side by electronic transitions and Raleigh scattering. Fluoride glass fibers have been traditionally prepared directly from the melt by using single crucible technique (3) or by drawing from a preform (4). Figure 2 illustrates typical apparatus. In order to reach the intrinsic limits of 10-2 to 10-3 db/km it has been determined that improvement in purity and quality of fluoride fibers are necessary. As an example of what limitations current processing techniques

provides, light scattering experiments on silica fibers by Tran (5) has indicated that crystallites formed in a glass fiber do attenuate thus limiting transmission below the theoretically attainable levels. There will be more discussion about this phenomena as it does provide rationale to consider containerless processing techniques in space as a possible route to obtaining high quality fibers.

Certain physical and chemical characteristics of the heavy-metal fluoride glasses favor their use over conventional glasses in applications requiring vitreous materials in bulk form. The relatively low glass transition temperature (Tg - 300 - 350 degrees C) allow the glasses to be formed at moderate pressures and temperatures. Also the chemical inertness of the glasses to corrosive fluoridating agents such as hydrogen fluoride and fluorine is especially useful in those kind of environments(6). In addition the mid-range infrared transmission characteristics of these fibers suggests a usefulness for transmitting optical power over short distances for applications in manufacturing such as cutting, welding, or drilling (7). Lasers which currently operate in the 1 - 5 micron range include iodine (1.315 microns), hydrogen fluoride (2.8 microns),

deuterium fluoride (3.8 microns), and carbon monoxide (5.0 microns). Other potential applications which are useful in the mid-range IR include surgical and thermal imaging.

The glass forming capabilities of the heavy-metal fluorides is not as good as silicate glasses. The question of glass formation is really a question of whether the viscosity is high enough to forestall crystalization while the melt is being cooled to the high viscosity region (8). Systems that form glasses under the slowest cooling rate are those with a high viscosity near their liquidus temperatures and whose viscosity increases rapidly below the liquidus. The heavy-metal fluorides are considered poor glass formers by these criteria due to their low viscosities at liquidus. Typically the viscosities are in the range .1 to 1.0 Poise at the liquidus of 600-700 degrees C, thus requiring relatively energetic quenching conditions. This characteristic of fluoride glasses needs to be taken into consideration if acoustic levitation furnaces are to be applied to these materials.

Knowledge of Tg and Tx is of interest for several reasons. In practical terms, Tg is a measure of the approximate upper-use temperature for a given composition. Annealing of the samples is then carried out at the lower temperatures. Temperature Tx defines a safe upper temperature for processing the melt if

devitrification is to be avoided. The quantity Tx-Tg then defines a working measure for the glass forming capability of the melt. According to Drexhage, it is desirable to have Tg-Tx as large as possible for fiber-pulling processing. In some sense it is also the resistence to devitrification. Figure 3 shows the typical relationship of Tg and Tx as obtained by Chung.(8)

The strong tendency toward devitrification during the melting and fabrication of fluoride glasses is partly due to the general features of their non-Arrhenius viscosity behavior. Hence heavy metal fluorides are very fluid near their liquidus and this low viscosity is retained upon cooling to Tg, at which point it increases rapidly. These materials thus have a very short working range, leading to rapid nucleation and growth not far from Tg.

Matecki (14) proposes that for many of the fluorides in use, the homogenous nucleation temperature is always above the glass transition temperature, thus implying that the generation of microcrystals proceeds at a diffusional time scale whenever the temperature of the melt is at or a little above Tg. They also suggest that this condition may be intrinsic throughout the entire glass-forming region; in others, optimization of

compositions is needed, so that glasses can be selected for compositions in which the homogenous nucleation temperature is above the glass forming temperature.

According to Drexhage's excellent summary on the heavy-metal fluoride glasses, the current state of knowledge regarding crystallization phenomena in the heavy-metal fluorides is more qualitative than quantitative. In addition to the homogenous nucleation effects discussed above glass samples sometimes contain isolated large crystals or groups of crystals that are readily visible at low magnifications under a polarizing microscope. The source of these crystals may be due to other process related effects, e.g. surface devitrification resulting from attack of the glass by atmospheric water or heterogenous nucleation on undissolved raw materials or impurities. Crystallization has been observed in rapidly cooled fluoride glass fibers, in which it results in optical attenuation because of light scattering. For example, Mitachi and Miyashita (15) observed scattering losses in gadolinium-containing fluorozirconates proportional to the second power of the wavelength. Such behavior is characteristic of scattering centers with dimensions comparable to the light wavelength. The strong tendency of the heavy-metal fluorides toward devitrification during the melting and fabrication of fluoride glasses is

partially accounted for by the general features of their viscosity temperature behavior. For example, fluorozirconate melts show a highly non-Arrhenius viscosity temperature dependence and are very fluid near their liquidus.

Several workers have examined the behavior of the viscosity fluorides near Tg. For example, the results of Moynihan,et.al. (16), determined the ΔH of one fluoride glass composition with another. His results indicated that since Tg is roughly an isoviscous point corresponding to 1013 Poise, the higher ΔH is near Tg, the more rapidly the viscosity drops to low values at temperatures above Tg and the more susceptible the melt is to devitrification. Figure 4 illustrates the viscosity-temperature behavior of vitreous heavy-metal fluorides. Similarly Figure 5 compares viscosity data for several glass forming systems, in addition to the fluorides.

A major thrust of the many programs dealing with heavy metal fluoride glasses tend to concentrate on the purity of the melt. As mentioned earlier (and shown in Figure 5) a significant impact upon the transmission capability of the fluorides is the presence of impurities such as OH, Eu and Fe. The typical fiber drawing apparatus used by many investigators (Figure 2, taken from Drexhage) requires a substantial amount of surface area for

contact between the containing walls and the glass melt during the drawing process. Hence, once purification of the starting materials has been performed, the purity of the melt may become contaminated again through this interaction with the container walls. Containerless processing does have promise in that once these contaminants have been chemically removed from the starting materials, pulling fibers from a levitated melt would insure that contaminants are not re-introduced into the fiber during the melting and pulling processes. Unfortunately combining acoustic levitation with space processing does not as yet provide all the easily obtained results.

Drehage has itemized several concerns in producing high quality fluoride fibers:

1. Proximity of glass transistion and crystalization temperatures.
2. Multiple components may increase light scattering.
3. Small crystallites observed in bulk samples.
4. High thermal expansion coefficients
5. Relativiely low draw viscosities.

As mentioned earlier, some proponents of space processing feel that item 3 will be minimized in space. Current knowledge about nucleation phenomena does not really explain why it should. Items 4 and 5 have been looked into by Weinberg (9) but to date the results are inconclusive. Some properties of several fluoride glass compositions are given in Table I as representative of these materials. (Taken from Drexhage, reference 1)

ACOUSTICAL FURNACE CONSIDERATIONS

In considering the concept of using acoustic forces to levitate molten glass for pulling fibers from the melt, several acoustic characteristics need to be considered before implementing such a system. Historically several workers have provided theoretical foundations for deriving expressions used to calculate pressure and force parameters in these types of experiments. (10) - (13)

The acoustic wavelength is important in that in a steady state pressure wave, the pressure nodes will dictate the position maintained by the molten glass. Since the wavelength of an acoustic wave is proportional to the density of the gas; i.e.

$$C^2 = \gamma \frac{RT}{m} = \lambda^2 f^2$$

c = velocity of sound

λ = wavelength

where

$$\gamma = C_p/C_v$$

giving for the temperature dependence of the acoustic wavelength:

$$A = \sqrt{\partial \frac{RT}{mf}^2}$$

Consequently the acoustic wavelength increases as the square root of the temperature. Calculations for two different gases argon and nitrogen provides useful information in deciding what kind of gas should be used to pressurize an acoustic furnace. Table II on the next page shows this calculation. One should also note the difference between a monotonic gas such as argon and a diatomic gas such as nitrogen. Figure 7 shows this relationship in a graphical form.

TABLE II. Wavelengths of air and argon gases at various temperatures and 22000 Hertz.

TEMPERATURE	AIR WAVELENGTH	ARGON WAVELENGTH
30	1.597	1.473
80	1.723	1.590
130	1.841	1.699
180	1.952	1.801
230	2.057	1.898
280	2.157	1.990
330	2.252	2.078
380	2.344	2.162
430	2.432	2.244
480	2.517	2.322
530	2.599	2.398
580	2.679	2.471
630	2.756	2.543
680	2.831	2.612

The acoustic pressures obtained in steady state fields has been studied by many workers. Most notable are King, Crum, and Yosioka and Kawasima. The expressions obtained by these scientists appear in the literature as:

$$F = \frac{V_o P_a^2 k_z \sin(2k Z)}{4\rho c^2} \left(\frac{5\delta - 2}{2\delta - 1}\right)$$

Obviously then the force of an acoustic standing wave would vary as inverse temperature in the gas supporting the standing wave. This relationship can be expressed as:

$$= \frac{V_o P_a^2 k_z \sin(2K Z)}{4\rho \lambda^2 f^2} \left(\frac{5\delta - 2}{2\delta + 1}\right)$$

Where V_o = equilibrium volume of droplet

P_a = steady state pressure

$k = 2\pi/\lambda$

ρ = density of air

$\gamma = \frac{\rho^*}{\rho}$ ratio of droplet density to density

f_z = acoustical frequency

EXPERIMENTAL

The experiments performed within the context of this report were performed using an acoustic leviatation furnace built by Intersonic, Inc.under another contract. Initial requirements were generated for the delivered acoustic furnace before the principal investigator became involved with the apparatus. Consequently a

number of features desired for experimentation with acoustic levitation of molten glass were not included in the system. Among these were power measurement, force or pressure calibration, fiber pulling from two opposite sides, and the ability to pressurize the furnace. We did attempt to implement some of this capability in the SSL Laboratory. A RMS power meter was purchased and used to monitor the power levels during operation of the furnace. It was used primarily for optimization of the acoustic power of the furnace during levitation of droplets.

Several features detrimental to materials processing experimentation in microgravity were inherent in this particular set of experiments. First this author felt that pulling fibers from an acoustically leviated melt in space was not really needed. Secondly the concept of pulling unidirectionally presents problems in that as the droplet loses mass, the surface area used to maintain the droplet's position in a steady state acoustic field drops off very rapidly. Hence one would be able to pull only until the actual fiber pulling force exceeded the acoustic forces felt by the droplet. At that point the remaining mass would be pulled onto the container walls. A better approach would have been to consider a concentrated heat source and then to design a furnace which would allow pulling with equal force from

two diametrically opposite directions. In this way there is always an opposite force of equal magnitude retaining the molten glass in the hottest part of the furnace.

The furnace used in this set of experiments was designed with one hole in the center so that a fiber could be pulled vertically from the top. A schematic of the furnace shows that four heater elements in the corners of the container provided the high temperature capabilty of the furnace. This poor design provided the following characteristics:

1. Heat losses through the aperture designed for fiber pulling made the center zone of the furnace the coldest part of the furnace. In low gravity environment, the lack of convection would reduce the amount of heat that would be lost; but in one G the hole on the top proved to be an enormous heat sink.

2. Temperature gradients in the furnace affect the acoustic properties of the gas in the furnace. These factors can be detrimental as the temperature of the gas increases.

3. Gas dynamics would indicate that higher pressures would improve on the stability of the levitation processes at higher temperatures . No provision was made for a higher pressure capability in the furnace. For space applications, this concept also can prove to be a problem.

These points are further discussed with the figures that illustrate the results obtained below.

Due to the difficulties associated with preparing fluoride glass samples from the respective components and then purifying them, a major investment in time and money, Dr. Etheridge decided that obtaining a sample from groups already involved in fluoride glasses would be the most expedient route to take. We did contact Dan Tran, then at the Naval Research Laboratories, and he was gracious enough to send us a sample to use in these early experiments. We utilized only this one sample in the experiments before the contract period ran out.

Toward the end of the experimental work performed here, a ten kilowatt carbon dixode laser was used to heat the glass samples. Needless to say this heating technique worked much better than the original heating elements. Unfortunately for space experimentation, the concept is not as yet cost effective.

RESULTS

The force measurements were observed by suspending a balance weight through the aperture at different positions. The data were taken at positions which varied at even distances. Since these distance differences had no relation to the acoustic wavelength of the furnace the plotted data bears no relation to the sine wave which is expected. However one can plot the function

and notice that the same type of plot is obtained. We were not able to obtain geometrical parameters of the suspended weight and come up with a suitable fit to completely match the experimental data. Since the molten glass will have a different characteristic (its a deformable body), we did not think that it was important at this stage. Figure 8 shows how the function above plots using

Lotus 1-2-3, while Figures 9, 10, and 11 show plots of actual data from the force balance measurements. Correlation of these data point through another program were good.

Another problem encountered with the experimental arrangement contained in this acoustic furnace was the inadequate thermal capability to melt a glass sample in the center. As mentioned earlier, the hole at the top just does not work well in a one G environment. Thermal mapping of the furnace was performed using a thermocouple as shown in Figure 12. The results are shown in Figure 13 with the lowest temperature in the center of the furnace. Needless to say we were not consistently able to melt any glasses using the furnace. In low G, one can anticipate that reduced convection of gases in a temperature gradient would reduce the problem. As a matter of course we did prepare the package for KC-135 experimentation, but since we could not melt fluoride glasses on the ground with the furnace, Dr. Ed Etheridge decided not to expend the funding required for KC flights. A matter of concern to the principal investigator was the consequences of experiencing nearly two G's during the parabolas of the KC-135 flights. It was felt that the heating capability of the provided heating elements would not be able to maintain

uniform temperature on a molten glass sample during such manuvers. Consequently we also felt that the KC-135 experiments would not be beneficial under such conditions.

We conclude these sets of experiments by using the 10 KW carbon monoxide laser in Dr. Etheridge's laboratory as a heating source with the acoustic levitator and had much better results in being able to pull fibers from an acoustically levitated melt. Figure 14 illustrate this arrangement. Since it was not flyable on either the KC-135 or Space, no other experimentation was performed.

CONCLUSIONS

Acoustic levitation at high temperatures is a new experimental domain for the space processing domain. More experimentation with fundamentals of fiber pulling processes need to be performed in ground based environments which can then be tranformed into space processing experiments once we have learned how to perform the experiments. Laser heating does work well on the ground. We have to learn how to tranform this technology cost effectively to space and then fiber pulling may be possible.

Acknowledgements

I wish to thank and compliment a number of students who performed well in this effort. Firstly Mr. Robert Bond for showing leadership in supervising some of the less experienced students, Ms. A. Ford, Mr. Bruce Mowery, and Mr. Mike Giuntini. Their participation in this project was educational and I believe we all learned something in trying to adapt simple objectives into space processing experimentation.

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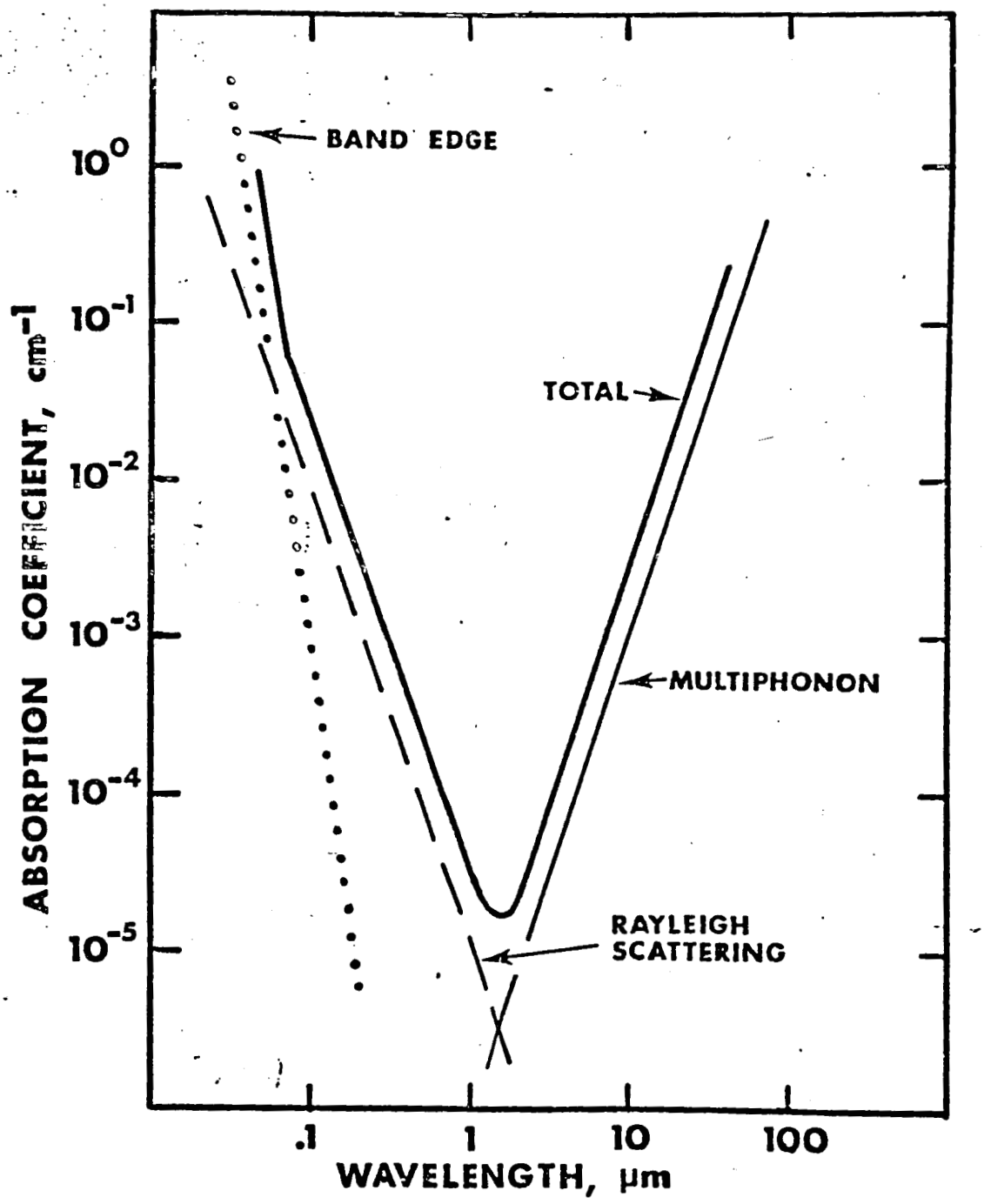
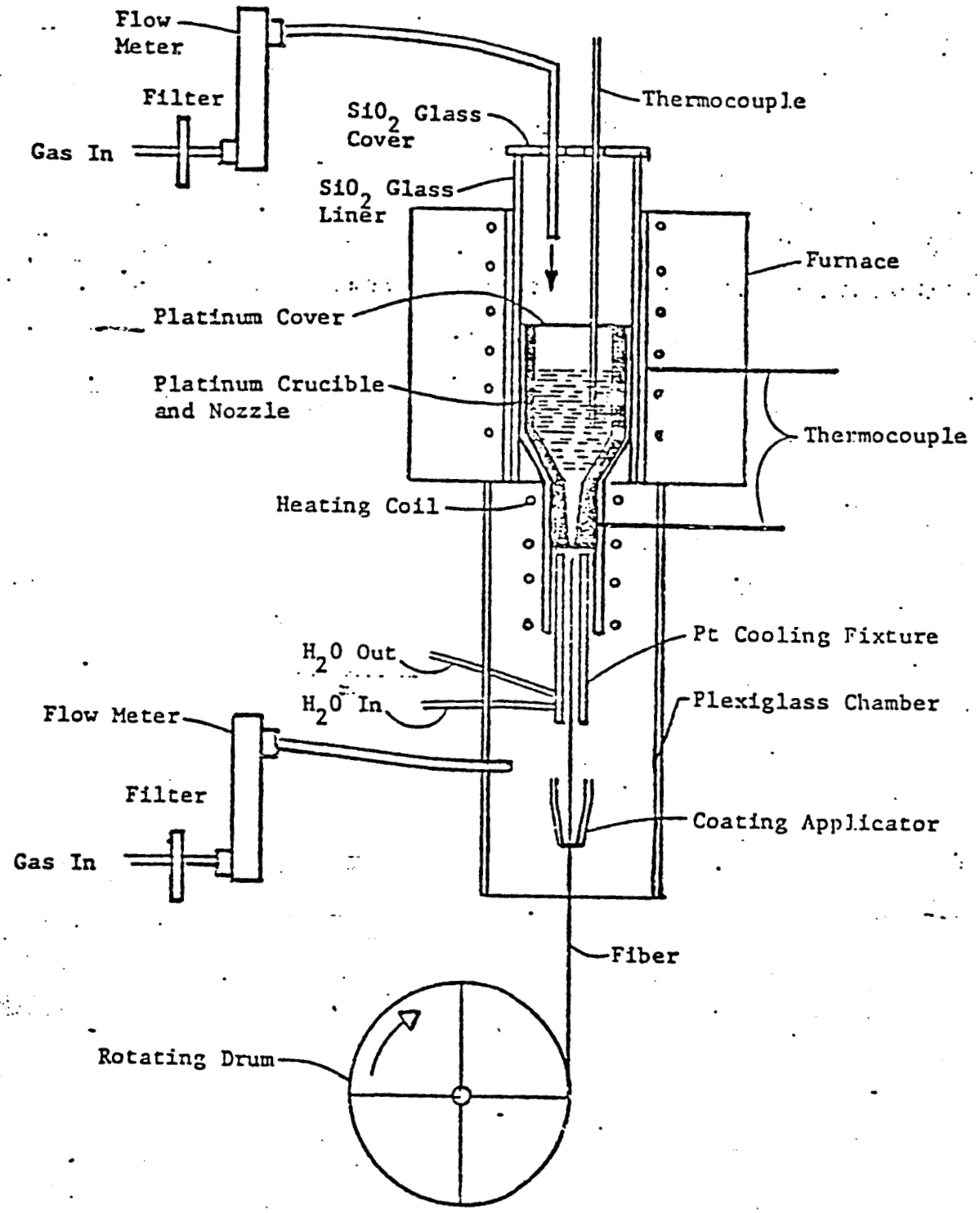
FIGURE 1.



THEORETICAL LIMITS ON FLUORIDE GLASS TRANSMISSION

FIGURE 2. ORIGINAL PAGE IS OF POOR QUALITY



TYPICAL GLASS DRAWING APPARTUS

FIGURE 3.

TYPICAL DIFFERENTIAL SCANNING CALORIMETER TRACE
SHOWING GLASS TRANSITION AND CRYSTALLIZATION TEMPERATURES

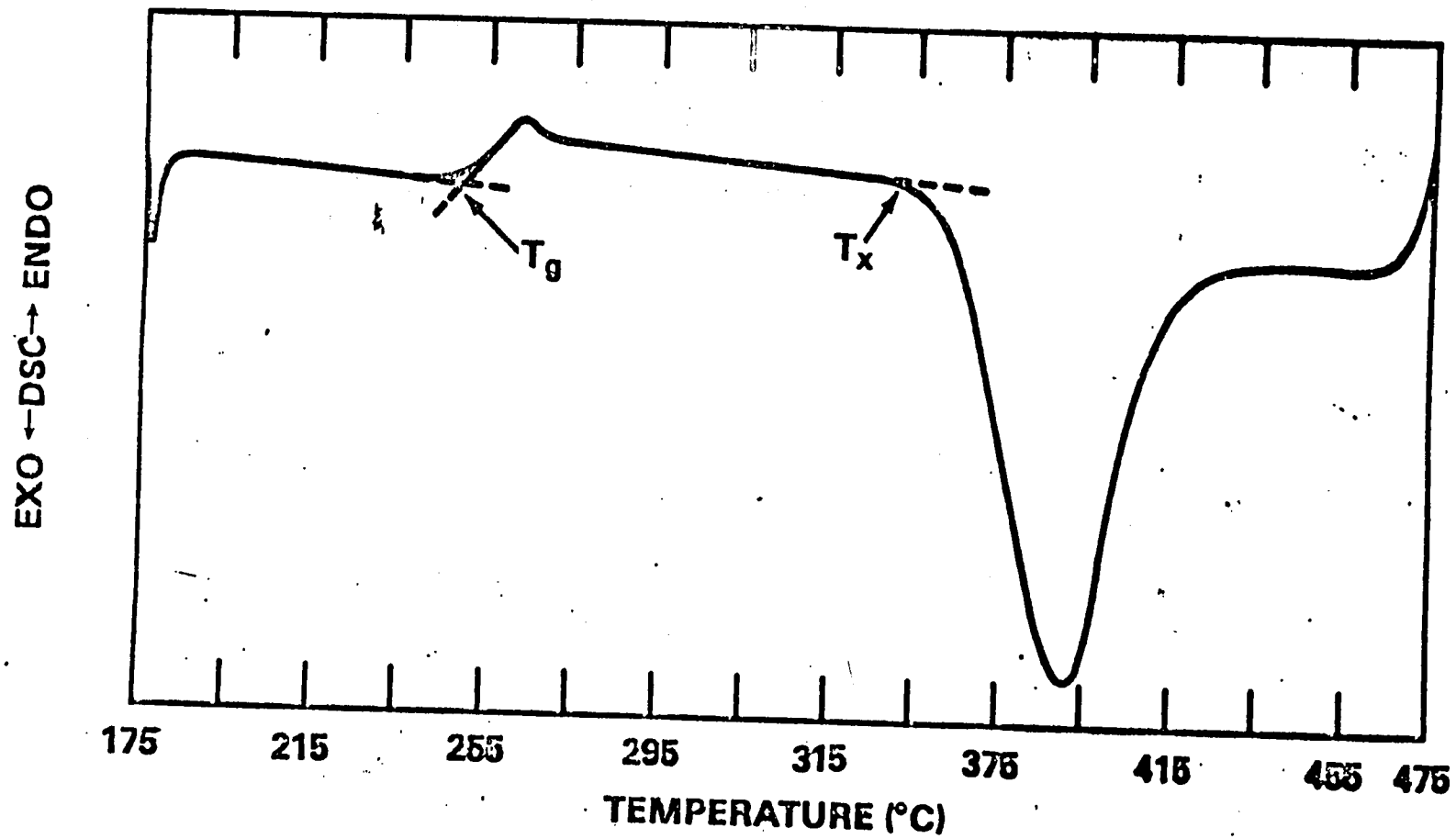


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FIGURE 4.

TEMPERATURE - VISCOSITY PROFILES
FOR SELECTED FLUORIDE GLASSES

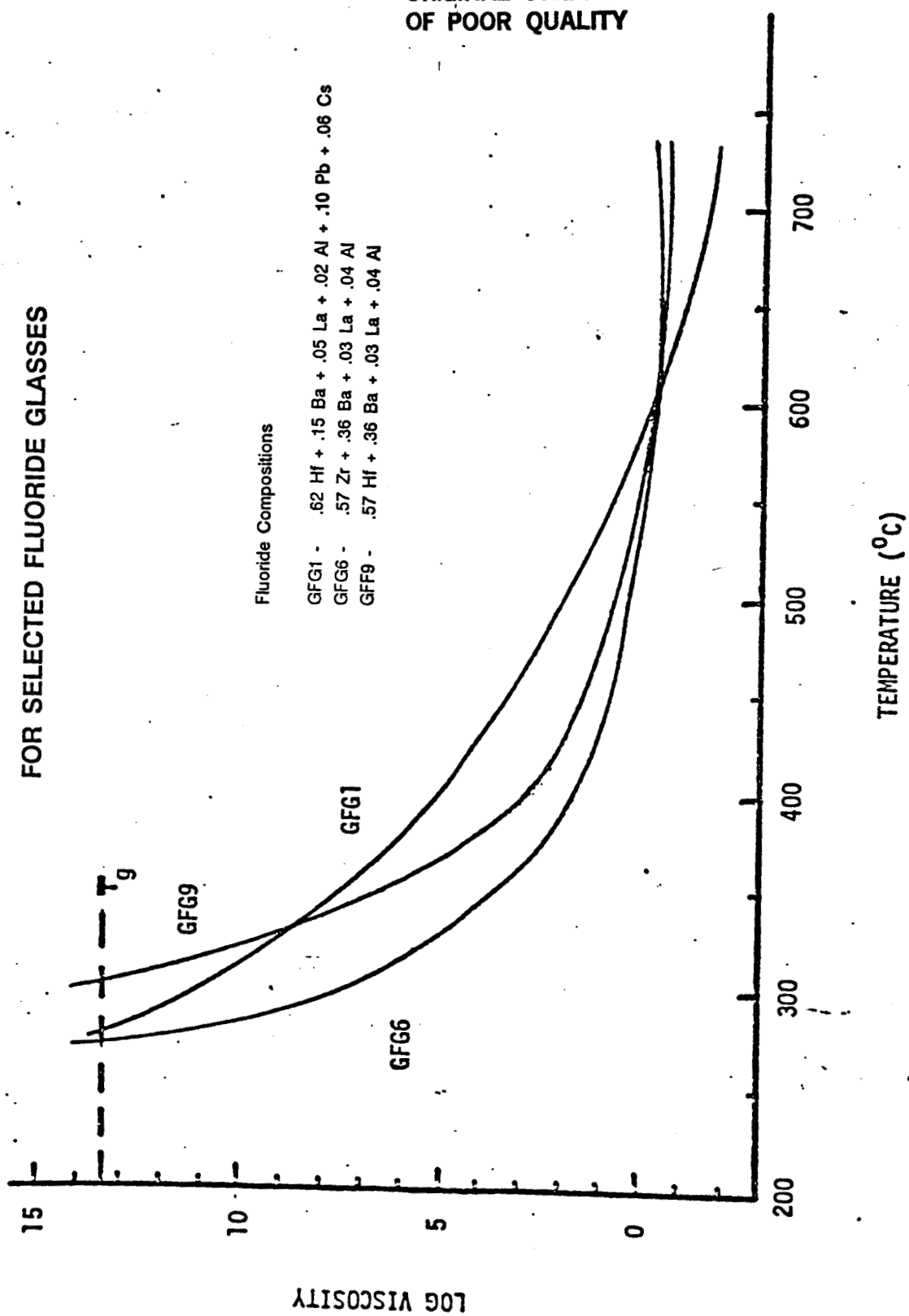
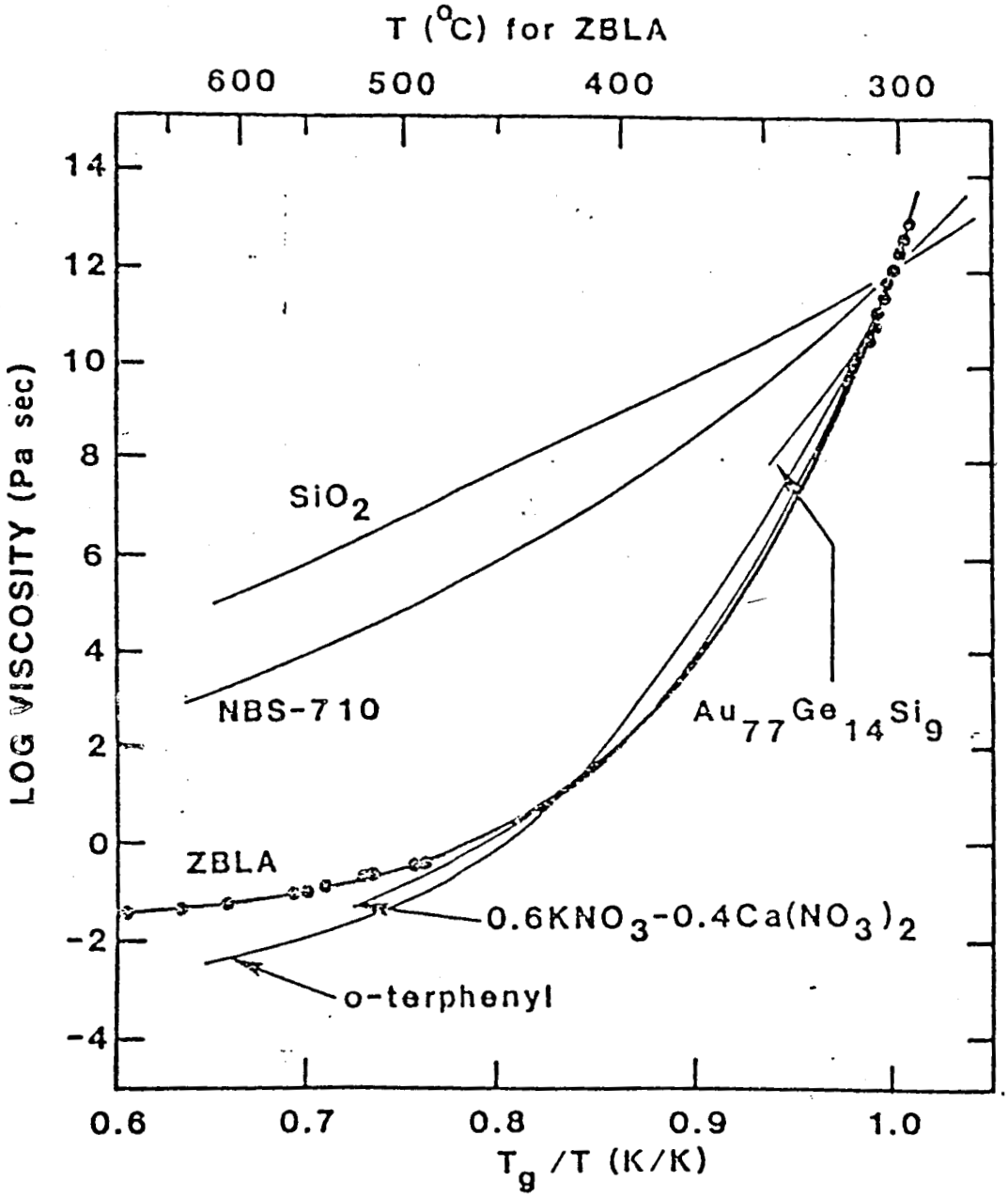
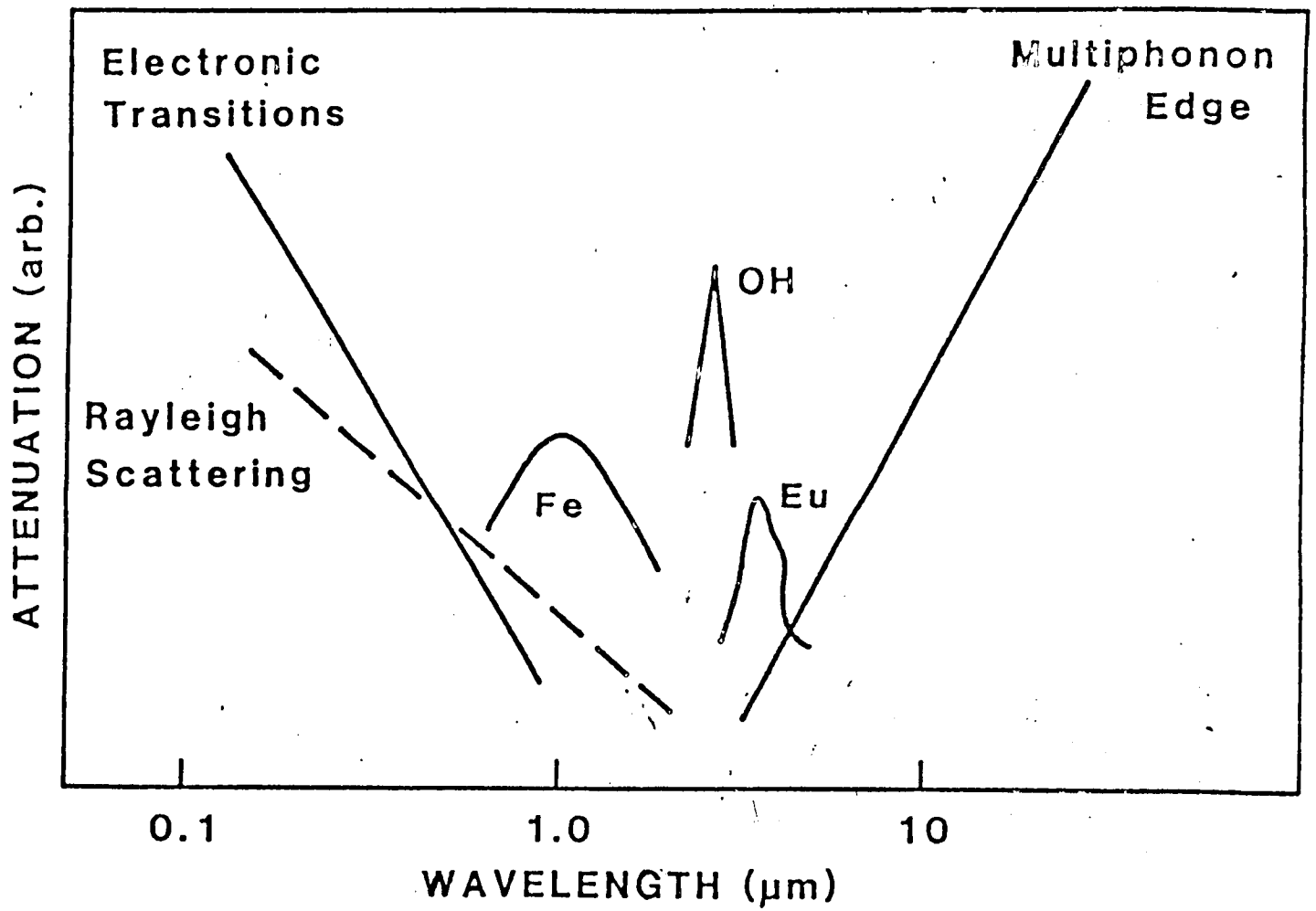


FIGURE 5.



Arrhenius Plots on Reduced Temperature Scale of Shear Viscosities of Different Glass Forming Melts

FIGURE 6.



Schematic Representation of the Mechanisms Responsible for Optical Absorption in a Hypothetical Vitrous Solid

TABLE I

COMPOSITIONS AND PROPERTIES OF SELECTED BINARY, TERNARY, AND QUATERNARY FLUOROZIRCONATE AND FLUOROHAFNATE GLASSES

Composition (mole %)	T_g (°C)	T_x (°C)	Density (gm/cm^3)	n_D	Reference(s)
$64ZrF_4$–$36BaF_2$	300	352	4.66	1.522	Almeida, 1980
$53ZrF_4$–$47ThF_4$	490	572	5.72	1.551	Matecki *et al.*, 1982a
$50ZrF_4$–$25BaF_2$–$25NaF$	240	300	4.50	1.50	Poulain and Lucas, 1978
$63ZrF_4$–$33BaF_2$–$4GdF_3$	310	390	—	1.529	Mitachi *et al.*, 1981c
$62ZrF_4$–$33BaF_2$–$5LaF_3$	306	380	4.79	1.523	Drexhage[a]
$62HfF_4$–$33BaF_2$–$5LaF_3$	312	395	5.78	1.514	Drexhage[a]
$57.5ZrF_4$–$33.75BaF_2$–$8.75ThF_4$	320	400	4.80	1.523	Poulain *et al.*, 1977
$57.5HfF_4$–$33.75BaF_2$–$8.75ThF_4$	319	396	6.19	—	Drexhage *et al.*, 1980a
$55ZrF_4$–$30BaF_2$–$15UF_4$	320	400	5.01	—	Aliaga *et al.*, 1978
$50ZrF_4$–$43ThF_4$–$7YF_3$	465	559	5.41	1.537	Matecki *et al.*, 1982a
$45ZrF_4$–$36BaF_2$–$11YF_3$–$8AlF_3$	334	425	4.54	1.507	Lecoq and Poulain, 1980a
$57ZrF_4$–$36BaF_2$–$3LaF_3$–$4AlF_3$	310	390	4.61	1.516	Drexhage[a]
$57HfF_4$–$36BaF_2$–$3LaF_3$–$4AlF_3$	312	400	5.88	1.504	Drexhage[a]

[a] Data for glasses prepared and characterized by the author and co-workers at RADC, Rensselaer Polytechnic Institute and Galileo Electro-Optics Corporation.

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FIGURE 7. Variation of Acoustic Wavelength with Temperature at 22000 Hertz for Air and Argon

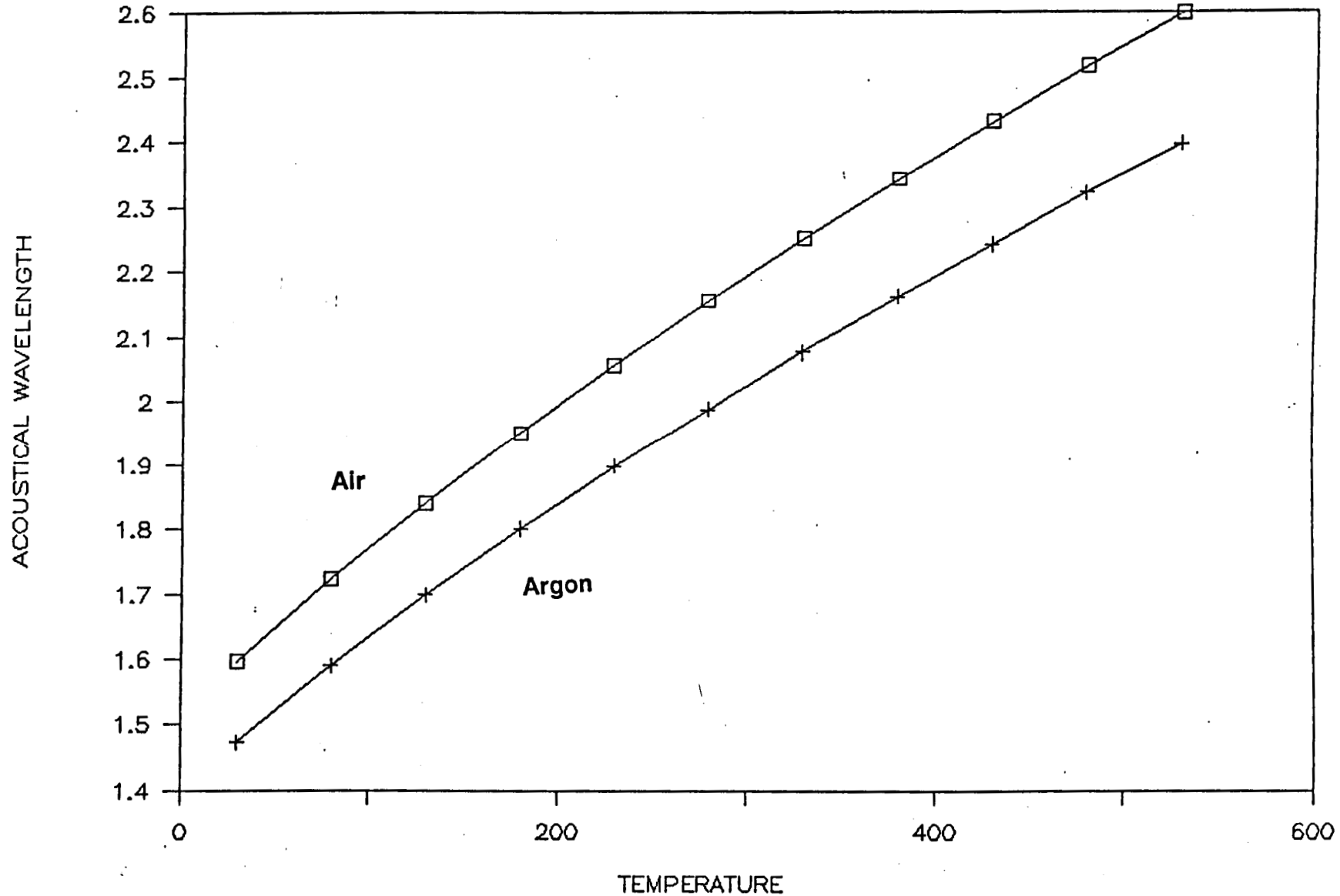


FIGURE 8. SIMULATED FORCE PROFILE

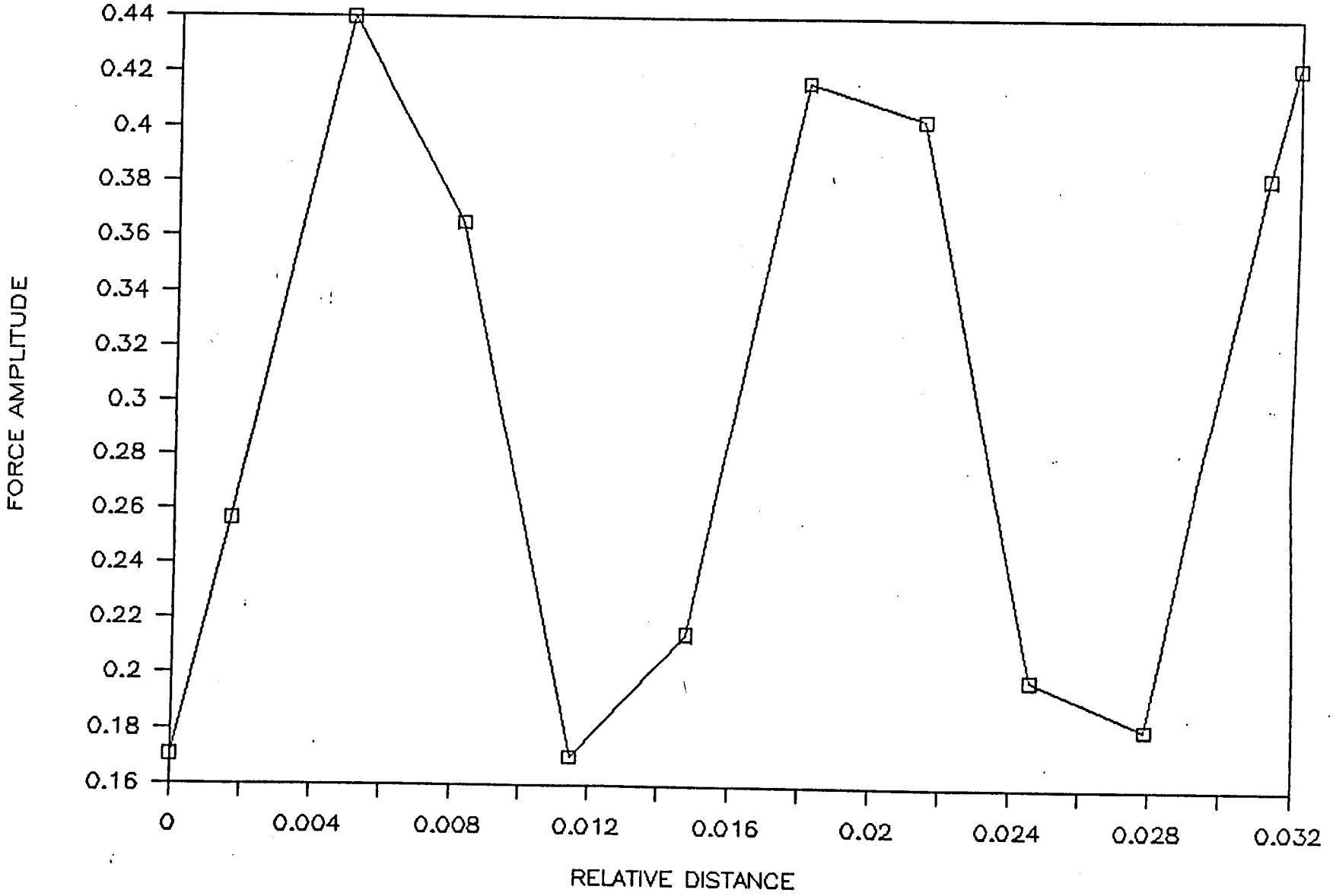


FIGURE 9. ACOUSTIC LEVITATOR FORCE

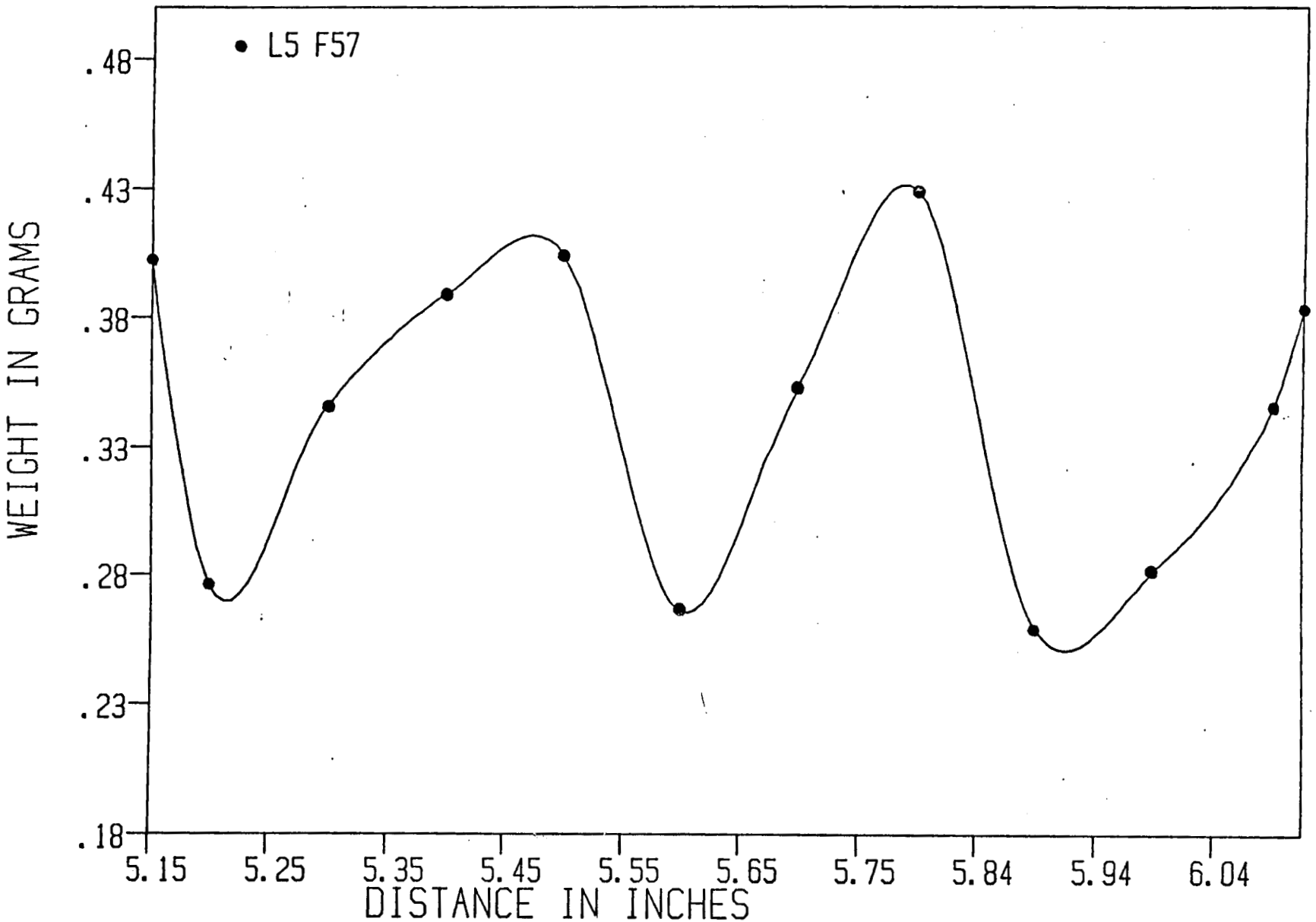
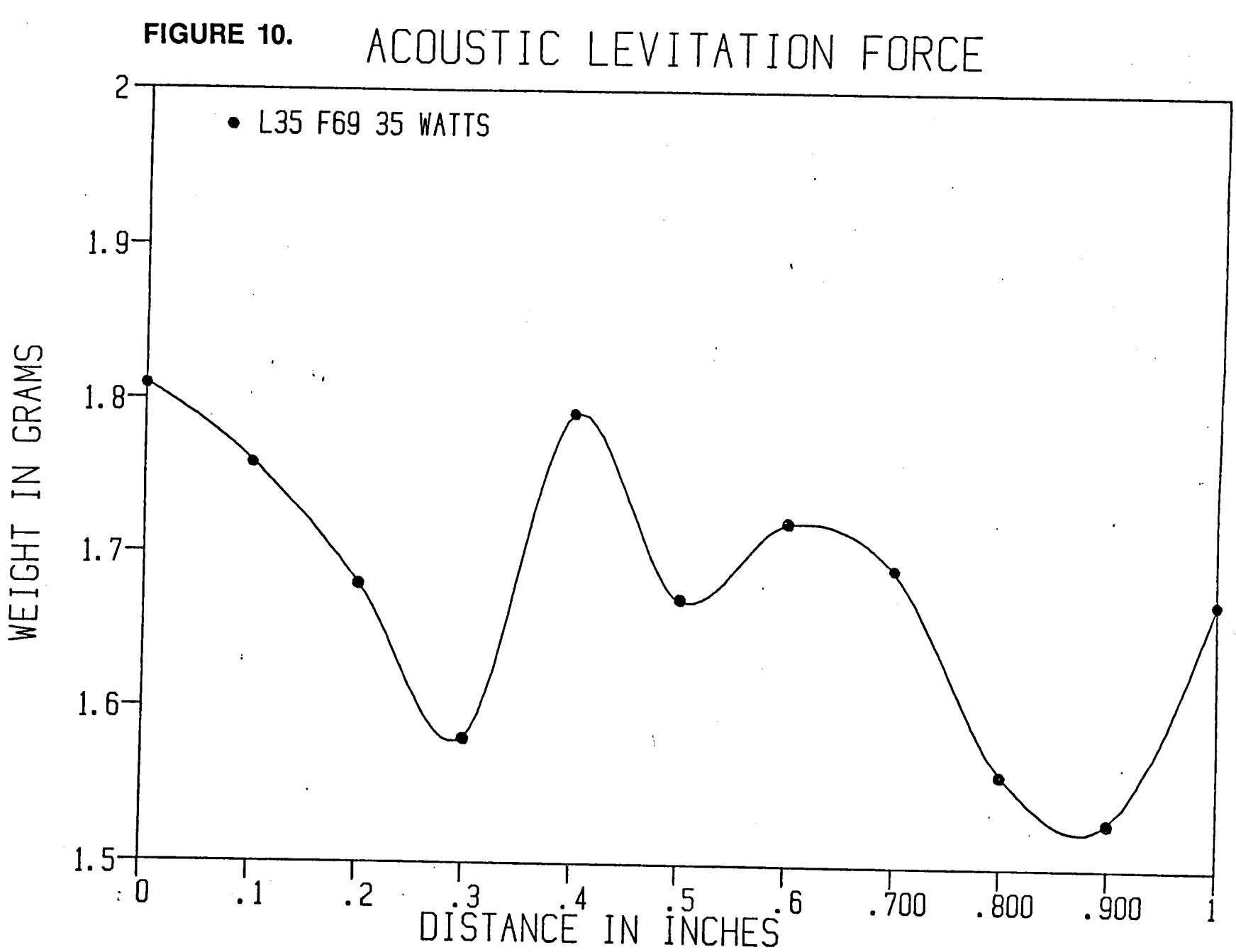


FIGURE 10.


ACOUSTIC LEVITATION FORCE
• L35 F69 35 WATTS
WEIGHT IN GRAMS
2
1.9
1.8
1.7
1.6
1.5
0
.1
.2
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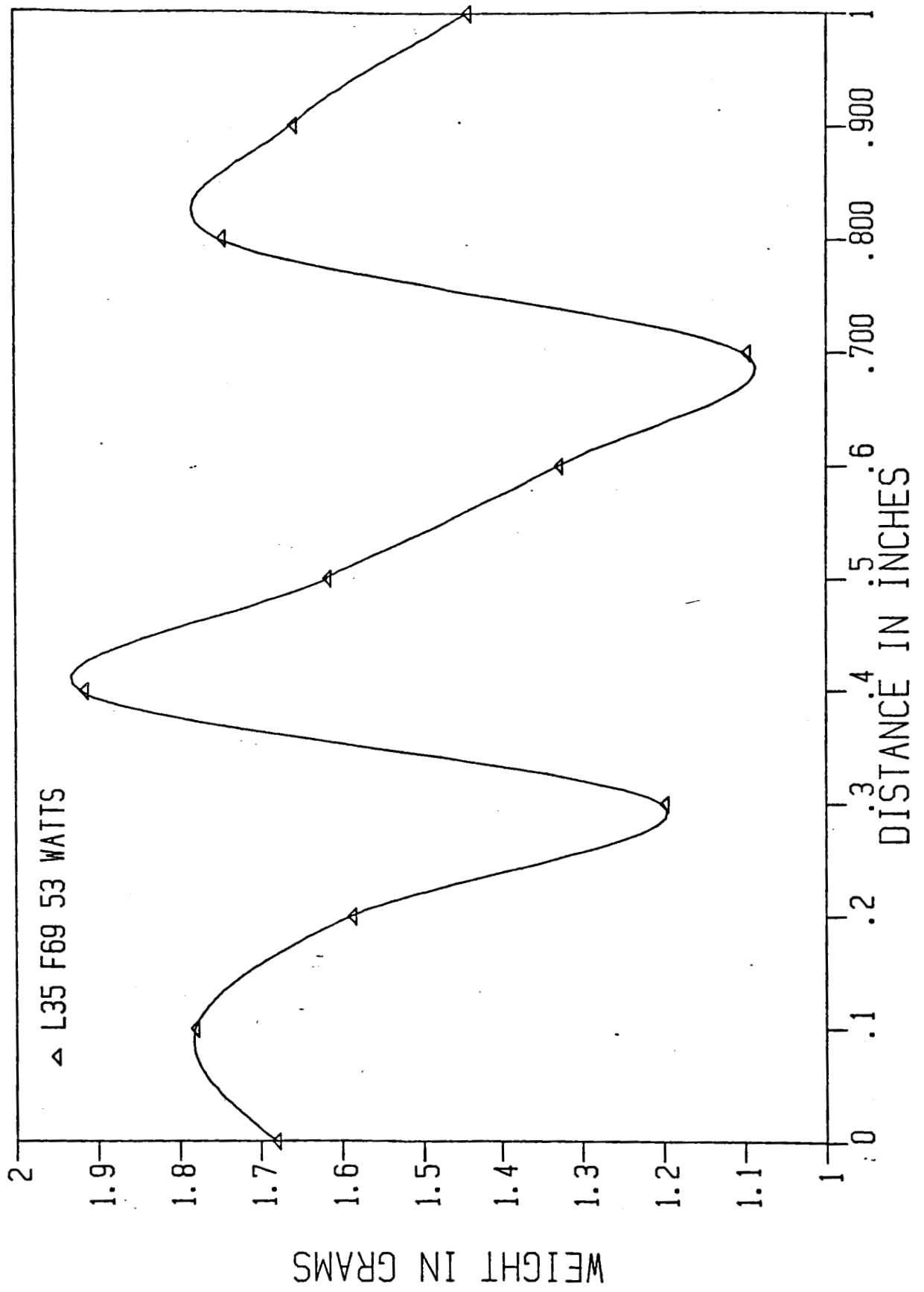
.4
.5
.6
.700
.800
.900
1
DISTANCE IN INCHES


ACOUSTIC LEVITATION FORCE
△ L35 F69 53 WATTS
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.700
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.900
1
DISTANCE IN INCHES

FIGURE 11.

FIGURE 12. EXPERIMENTAL ARRANGEMENT FOR FORCE MEASUREMENT

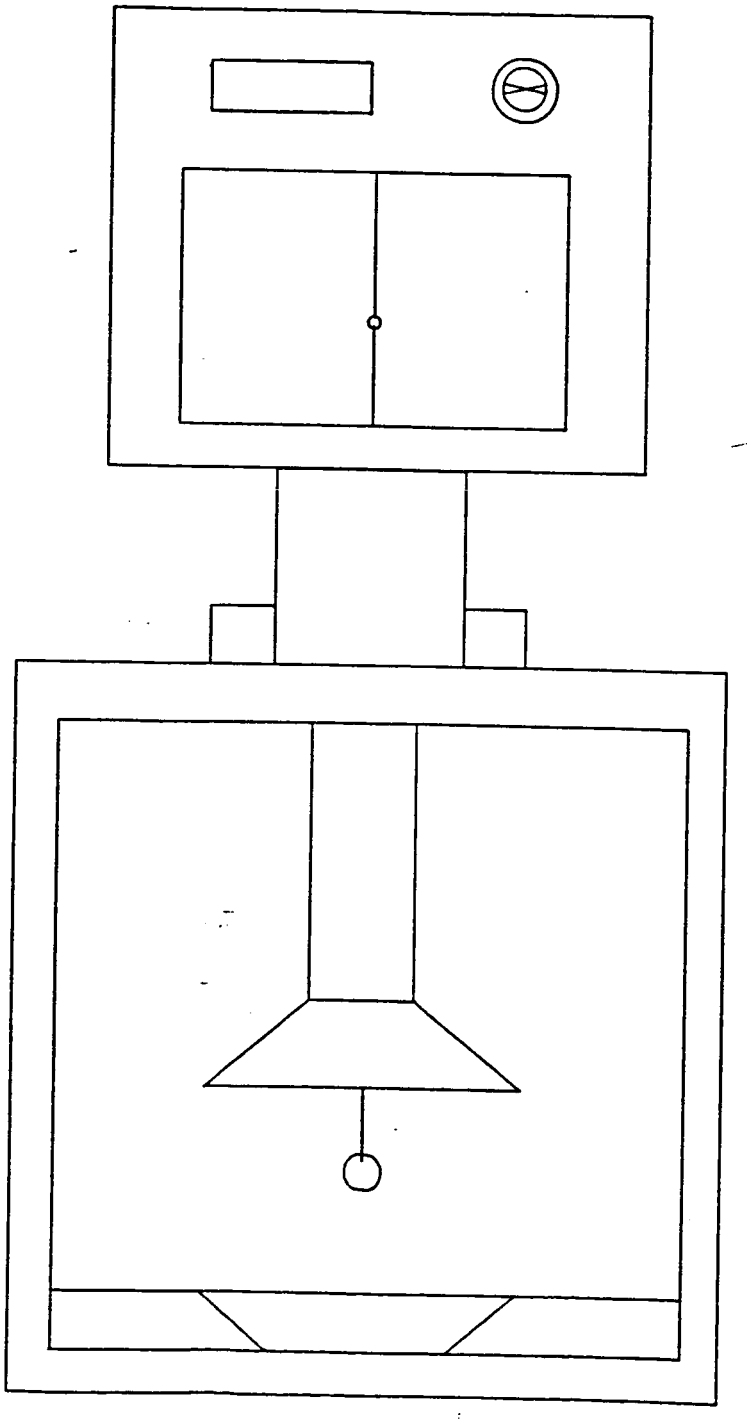


FIGURE 13.

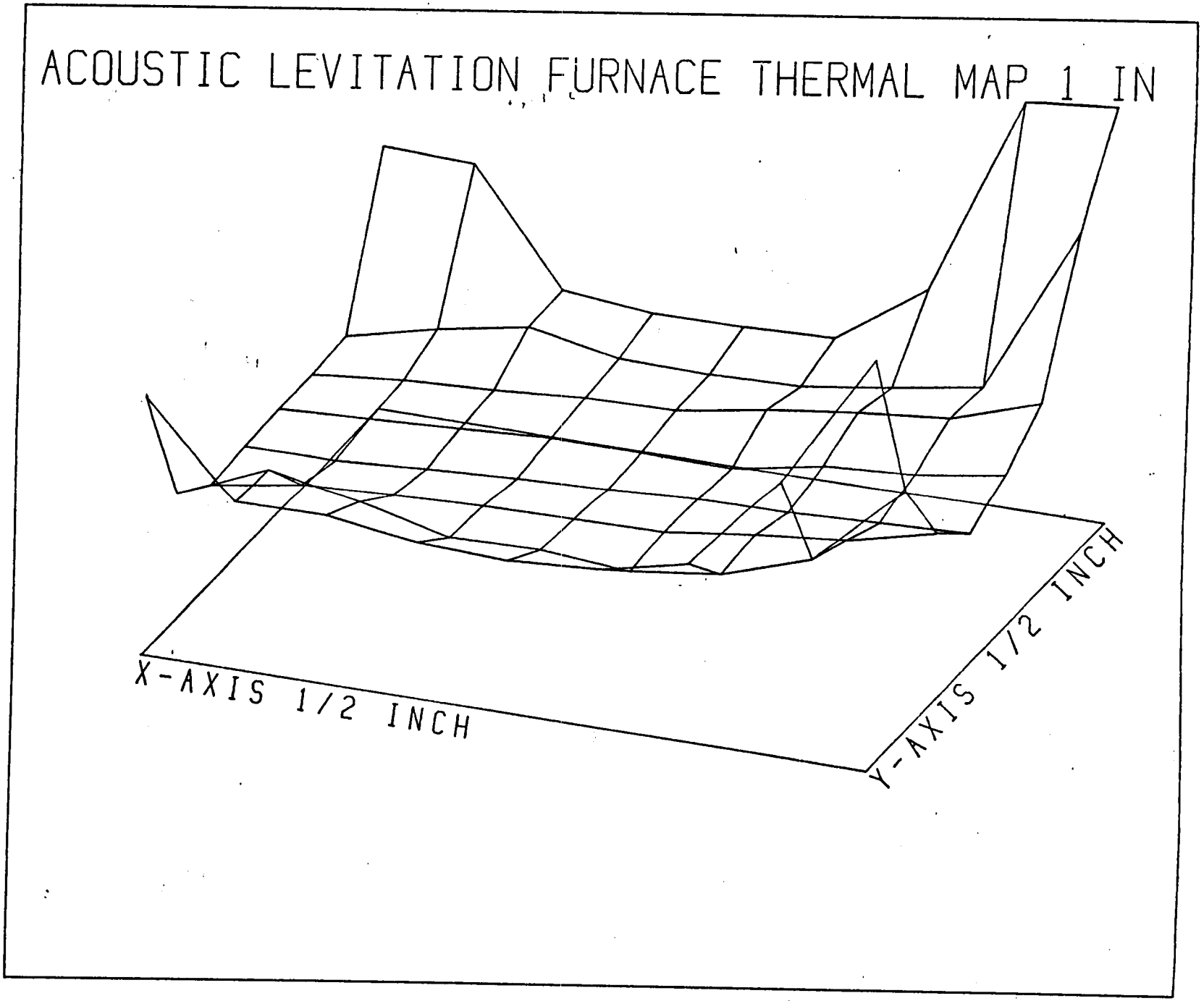
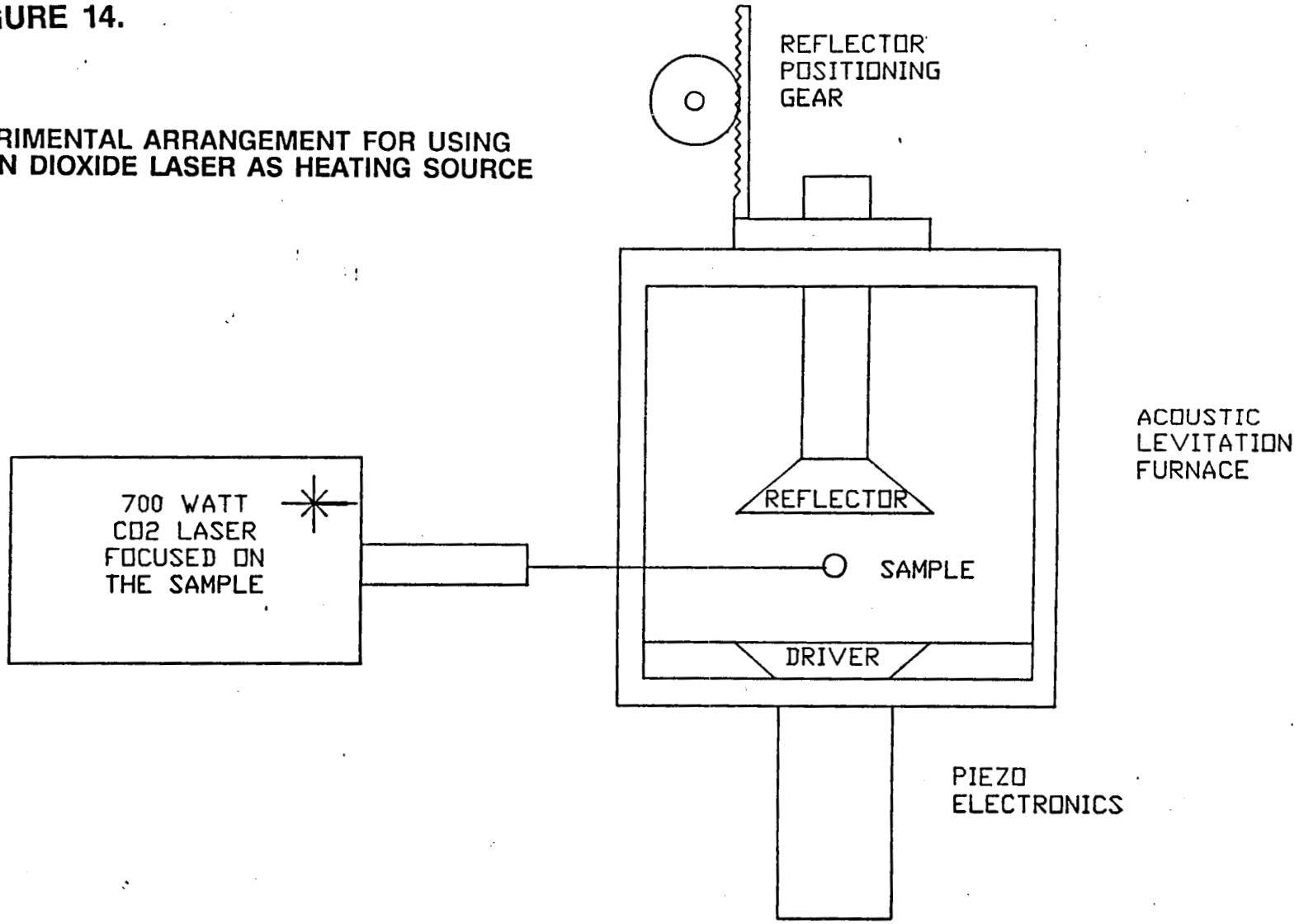

ACOUSTIC LEVITATION FURNACE THERMAL MAP 1 IN
TEMPERATURE IN C
X-AXIS 1/2 INCH
Y-AXIS 1/2 INCH

FIGURE 14.

EXPERIMENTAL ARRANGEMENT FOR USING CARBON DIOXIDE LASER AS HEATING SOURCE



FINANCIAL STATUS REPORT

CONTRACT NAS8-35978

Total Cumulative Costs incurred as of 3-25-86 (date) $36,333.67

Estimate of cost to complete 0.00

Estimated Percentage of Physical Completion 100%

Statement relating the Cumulative cost to the percentage of physical completion with explanation of any significant variance:

If you have questions concerning this statement, they may be addressed to Karen Allison, 895-6421.